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IM/EM Technology Implementation in the 21st Century

BDNPA/BDNPF SHOWS LONG-TERM AGING STABILITY

PRESENTED TO NATIONAL DEFENSE INDUSTRIAL ASSOCIATION

BY

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INTRODUCTION

In about January of 1946, the Department of the Navy, Office of Naval Research funded a significant effort to develop a smokeless propellant based on polynitro-containing organic compounds at GenCorp Aerojet. Documentation of this Navy-funded effort, extending into about 1963 at Aerojet, can be found in a number of reports.[1] Although this work was originally classified as confidential, it was officially declassified by order of the Department of the Navy, Office of Naval Research (ONR:426:RLH:10, April, 1961 and EO11652, December, 1972). In 1994, the Department of the Navy and Aerojet, by way of letters of authorization, granted the co-author, Mr. Renato Rindone, publication rights for all of the work done under this contract. This research program resulted in the discovery of new synthetic routes and manufacturing methods for the preparation of *geminal* dinitro compounds. Most notably, a high energy, two component nitroplasticizer, composed of a 50/50 weight % eutectic mixture of bis(2,2-dinitropropyl)acetal [BDNPA] and bis(2,2-dinitropropyl)-formal [BDNPF], referred to as A/F, was prepared and successfully used in the Polaris missile system.

More recently, the U.S. Army Tank-automotive and Armaments Command – Armament Research Development and Engineering Center (TACOM-ARDEC) Explosives Research and Technology Team has found it advantageous to use the energetic plasticizer A/F in explosive formulations. The energetic plasticizer, A/F, allows for the development of explosive compositions that are less sensitive to initiation by outside stimuli while maintaining operational performance. A/F is successfully used in thirteen (13) of the twenty-four (24) recognized Picatinny Arsenal Explosives (PAX) formulations. The most well known being Picatinny Arsenal Explosive 2A (PAX-2A), the Army's less sensitive high explosive replacement, suitable for current and future weapon system's high performance warhead applications.

The U.S. Army, TACOM-ARDEC developed the PAX-2A explosive in 1989 as an alternative to the more sensitive HMX (i.e., LX-14, PBXN-5) and RDX (i.e., Comp-A3, Comp-A5) based high explosives. The composition of PAX-2A is 85% HMX, 9% A/F and 6% CAB. It is a pressed, plastic bonded explosive. PAX-2A has approximately 10% less HMX than LX-14, but makes use of the energetic plasticizer, A/F, to maintain energy output. Based upon tests conducted for its interim hazard qualification testing, PAX-2A has proven to be much less sensitive than LX-14 and Comp-A5.

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[1] *Research in Nitropolymers and Their Application to Smokeless Propellants* (and Supplements), Navy Contracts: NOas 7968, NOas 8446 T.O.2. N7-ONR-462, N7-ONR-46208, NOas-53-618c, NOas-54-399c, NONR-2655(00)

Like most chemical products, a major concern about A/F is its shelf-life. This concern about the shelf-life of A/F has been a topic of discussion since A/F was first manufactured in the late 1950s and early 1960s. Depending on the manufacturing and quality control methods used in the preparation of A/F, the shelf-life was found to vary widely, ranging from about one year to as much as 30 (calculated) years in duration. Many factors are now known that effect the shelf-life of A/F including the acid content, type and concentration of impurities, type of stabilizer used and the storage temperature.

Historically, a major concern with storing A/F at ambient conditions has been in determining how long it would take before the "in spec" A/F deteriorated to a point where it was no longer acceptable material. This is an important consideration because weapon formulations containing A/F probably have a service-life that is in some way related to the A/F quality. Therefore, predicting the stability of A/F would be a useful tool in determining the serviceability of the munitions containing the A/F. One technique used to observe the A/F stability during storage is to determine the "acid number" at periodic intervals. After A/F passes the initial acceptance tests, tracking the quality of A/F during storage is done using acid number determination as a guide. It is known that if A/F deteriorates during storage, the acid number has a tendency to increase. The goal of this presentation is to show that A/F made with improved processing technology removing adverse impurities formed in the process will result in a product that has an exceptionally long shelf-life.

BACKGROUND AND HISTORY

In addition to the Aerojet effort, investigators at the Office of Naval Research, Purdue University and Ohio State University, also gave major contributions leading to the development of energetic nitro compounds. After about 1962, the work done by K. Baum at Fluorochem, M. Kamlet at NWC-Silver Springs, H. Adolph at NSWC-Indian Head, M. Frankel at Rocketdyne, H. Feuer and N. Kornblum at Purdue University, G. Olah at The University of Southern California, A. Nielsen at NWC-China Lake and J. Boyer at the University of New Orleans has greatly expanded the technology to prepare aliphatic and heterocyclic nitro and polynitro compounds. For a deeper

understanding of this chemistry, the reader is referred to the literature, replete with articles, reviews and texts on the chemistry of nitro compounds[2,3,4,5,6,7,8].

In the early Navy program effort, the outcome was the development of two different chemical processes to prepare A/F, one by Aerojet (Sacramento) and the other by the Navy (Indian Head). Both processes used nitroethane as the starting material and both processes prepared 2,2-dinitropropanol (DNPOH) as the key intermediate. The Aerojet process[9] used an improved ter Meer[10] reaction to prepare the DNPOH. The Navy process used oxidative nitration technology developed by Kaplan and Shechter[11] to prepare the DNPOH. The Aerojet process was scaled-up to continuous-flow production plant equipment in 1962 and the Navy process was scaled-up to batch size production plant equipment a little earlier. Although it was not understood at that time of production, the quality differences of the A/F from the two different processes played a significant roll in the shelf-life of the A/F and its propellant and explosive formulations.

There were advantages and disadvantages for both processes. The Aerojet continuous process was designed to manufacture about 1 million pounds/year of A/F using six processing steps. The overall A/F yield of this process was about 45% to 50% and the number of process impurities was considerably higher than the impurities formed in the four-step oxidative nitration process. The oxidative nitration process had a higher overall yield, of about 60%, but because the process used silver nitrate and because the individual components, BDNPA and BDNPF, were recrystalized before blending, it had a significantly higher operating cost and the A/F productivity was only a fraction of the Aerojet process. A reaction scheme for the ter Meer process is shown in *Figure I* and a reaction scheme for the oxidative nitration process is shown in *Figure II*.

Propellant for the Polaris A3 First Stage Rocket Motor was made using A/F prepared with the ter Meer process and the oxidative nitration process. Studies by the Navy and Aerojet early in the

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2 F.G. Noble, Jr., *et. al.*, *Chem. Reviews*, **64**(1), 19-58 (1964)
3 H. Feuer (editor), S. Patai (series editor), *The Chemistry of the Nitro and Nitroso Groups*, Part 1 (1969); Part 2 (1970), Interscience Publishers
4 S. Patai (editor), Supplement F, *The Chemistry of Amino, Nitroso, and Nitro Compounds and their Derivatives*, Parts 1 and 2, (1982), John Wiley & Sons
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6 E. Breuer, H. G. Aurich, A. Nielsen, *Nitrones, Nitronates and Nitroxides* (1989) John Wiley & Sons
7 A.G.M. Barrett (guest editor), *Tetrahedron*, “Nitroalkanes and Nitroalkenes in Synthesis”, **46**(21) (1990)
8 G. Olah, *et. al.*, *Nitration, Methods and Mechanisms*, (1994), VCH
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10 E. ter Meer, *Ann*., **181**, 1 (1876)
11 R.B. Kaplan, H. Shechter, *J. Am. Chem. Soc.*, **83**, 3535 (1961)

FIGURE I

A/F PREPARED BY THE TER MEER PROCESS

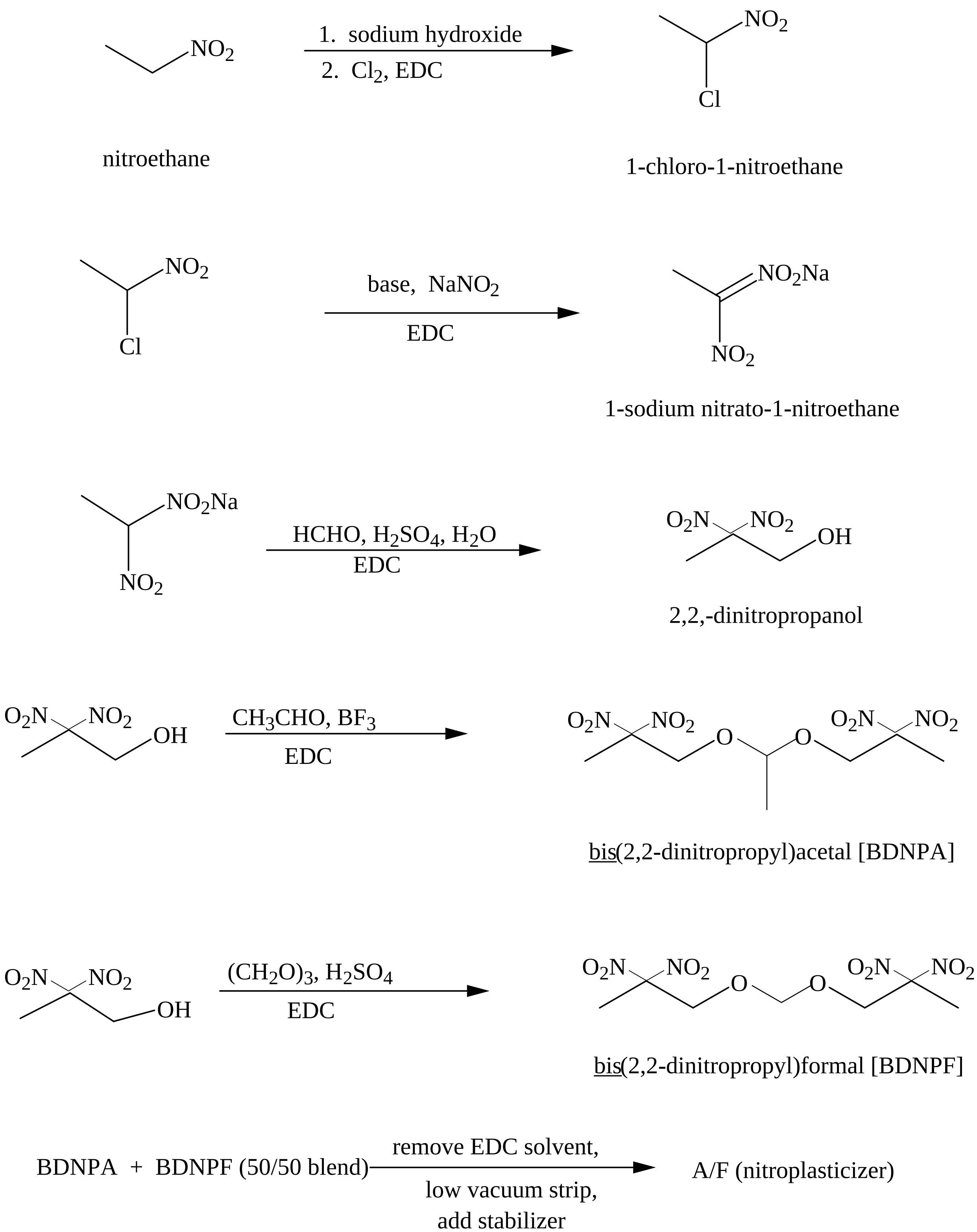
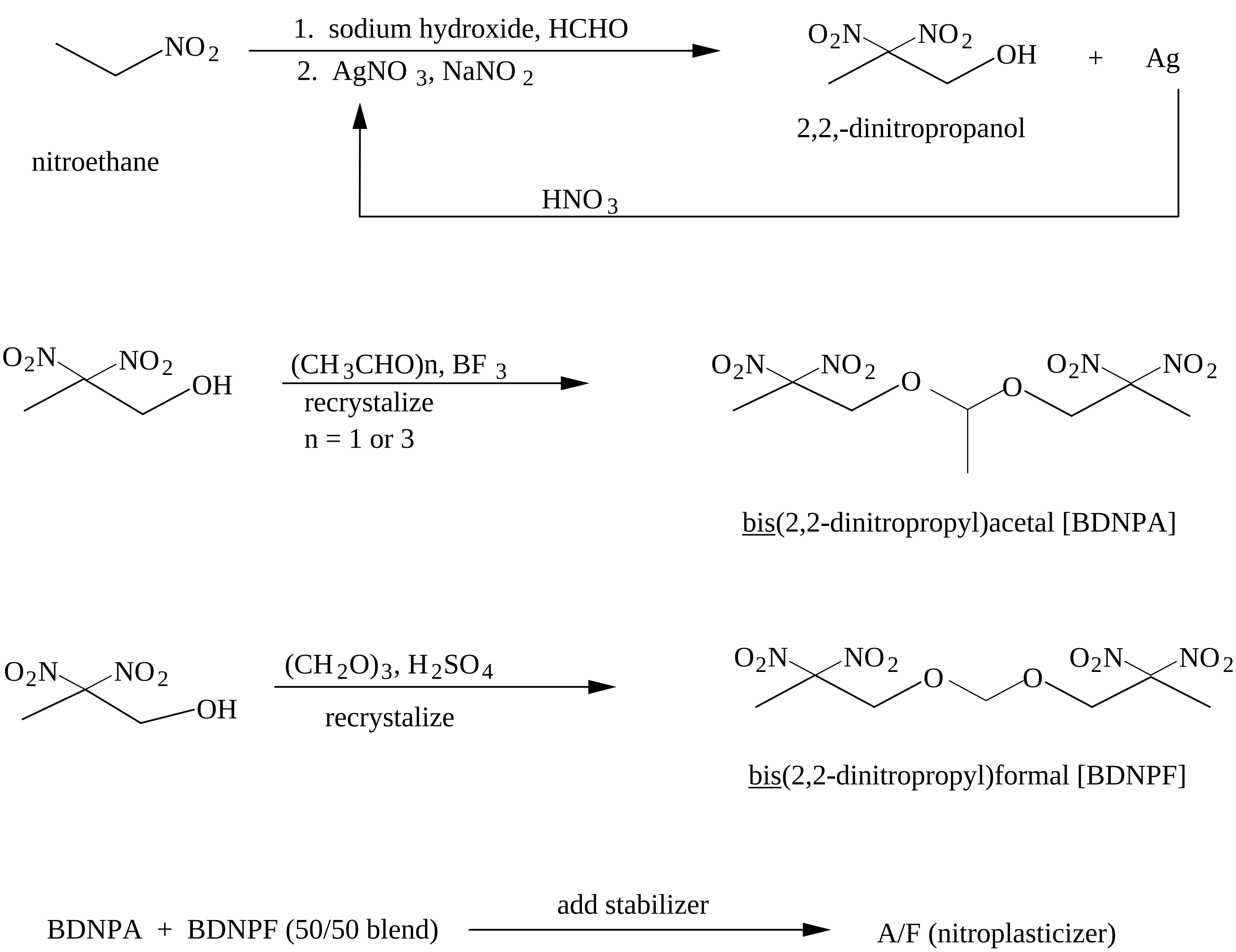


FIGURE II

A/F PREPARED BY THE OXIDATIVE NITRATION PROCESS



program showed that A/F made by the ter Meer process was inherently less stable than the A/F made using the oxidative nitration process. This was very interesting because the Aerojet A/F passed all of the product specifications that were in place at the time of manufacture. The original specification tests (WS 1141A) and limits are shown below:

Test	Limits
Acid Number, mg KOH/g	0.5 Max
Water Content, %	0.05 Max
N_d^t, 25° C	1.462 to 1.464
Density, g/ml, 25° C	1.383 to 1.397
BDNPA Content, wt. %	45.0 to 55.0
BDNPF Content, wt. %	45.0 to 55.0
PBNA Content, wt. %	0.08 to 0.18

By today's quality control standards, it is easy to see why there could be a substantial difference in the A/F made by the two processes. In the early production of A/F, the quality of the product from both processes was controlled by specifications that were limited to physical property measurements, such as density and index of refraction, and unspecific chemical analysis such as acid number determination and IR analysis of the C-NO_2 or C-O-C absorption bands. The earlier specifications were not specific enough to identify and directly control the concentration of the process impurities. At that time, sophisticated methods to identify process impurities were not possible because analytical techniques, such as capillary chromatography, hplc or e/m spectroscopy, were not fully developed. As a result, by the mid 1970s, at least 30,000 pounds of A/F made by the ter Meer process was found to be out of specification. In particular, after storage for a few years, this material had an extremely high acid number, ranging from about 0.6 to almost 5 mg KOH/gram-sample.

To rectify this problem, Aerojet developed a simple purification process for the "out-of-spec" A/F that greatly improved the A/F stability in accelerated aging tests (initially conducted at about 80° C) compared to previous lots of A/F made at Aerojet. In comparison to the A/F made by the oxidative nitration process, which was considerably more pure because undesirable impurities were removed during the recrystalization step, the Aerojet purified A/F showed it was more stable (i.e., purer) in the accelerated aging test. The reason for this improvement in stability was not immediately known, however, the vastly improved A/F stability gave cause for further studies to be conducted at Aerojet in the mid 1980s and early 1990s.

Clearly, the process conditions and equipment used to manufacture A/F played a critical roll in the resulting product quality. The role that the impurities played on A/F stability turned out to be far more significant than originally thought. Later laboratory studies showed that once these impurities were removed, there was no difference in aging stability of the A/F made by either the ter Meer or the oxidative nitration process.

TECHNICAL DISCUSSION

Laboratory studies conducted at Aerojet[12] have proven that the aging stability of A/F is primarily a function of its impurities. It follows that propellants made from A/F of questionable quality are more likely to show poor shelf-life than those made with a better quality A/F.

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[12] Presented to Dr. H. Adolph (NSWC Indian Head) and Dr. Kurt Baum (Fluorochem Inc., Azusa, CA), at Aerojet on November 8, 1990. Work done by Mr. James Barnard, et. al.

As a direct result of the aging problems experienced with the poor quality A/F, a new specification test method called "Accelerated Aging" was added to the A/F Weapons Specification (WS 1141A) for A/F produced from about 1983 to 1993. Although this test was not a specific test to detect impurities responsible for poor aging, it was a simple test and very suitable to determine the overall aging integrity of the A/F. In this test, a sample of A/F was maintained at 104° C to 105° C for a period of seven days and the acid number of the aged A/F was determined. If the acid number was greater than 0.5 mg KOH/gram-sample, the product was out of specification and not acceptable. This test was used as the basis to determine the impurity effects on A/F stability. Using capillary gas-liquid chromatography (glc), a number of unknown impurities were found to be present in the A/F. Of these, six major impurities were in concentrations ranging from about 0.1 to about 5 %. These impurities were isolated and identified using NMR, IR and mass spectral analytical techniques. These six impurities are listed in *Figure III* and a glc trace is shown in *Figure IV*.

The most significant impurity affecting the aging stability of A/F was found to be 2,6-dimethyl-4-(2,2-dinitropropoxyl)-1,3-dioxane (Compound V). The critical discovery about this impurity was that acid contamination was also needed for decomposition to occur. It was shown that both a source of acid (catalytic amounts) and Compound V needed to be present together before acid generation (i.e., increase acid concentration due to decomposition) would take place on aging. It was shown that in the absence of catalytic amounts of acid in the A/F the effect of the Compound V on decomposition was nearly non-existent. In fact, with greater than 99% confidence, the studies showed that the build-up of acid content in the A/F on aging was directly proportional (nearly mole for mole) to the concentration of Compound V provided that another acid impurity was also presenting the A/F. Neither BDNPA nor BDNPF was decomposing under accelerated aging conditions. Only the decomposition of Compound V was causing the increase of acid content. The correlation of different concentrations of acid impurity and Compound V on the probability of the A/F passing the accelerated aging test specification is shown in *Figure V*[13].

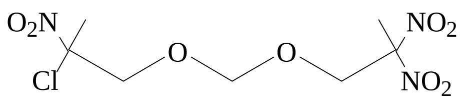
Compound V was formed due to the presence of aldol (a condensation product of acetaldehyde) in the BDNPA. This material was formed only in the BDNPA reaction. The chemical reaction scheme for BDNPA and the formation of the Compound V impurity is shown in *Figure VI*.

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[13] The Accelerated Aging Test required that A/F be heated at 104° C to 105° C for a period of one week and not exceed the 0.5 mg KOH per gram sample acid number

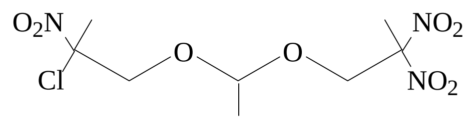
FIGURE III

ISOLATED A/F IMPURITIES FORMED IN THE TER MEER PROCESS



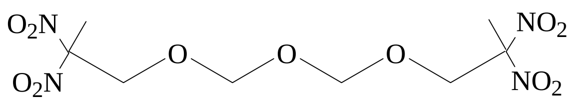
Compound 1

2-chloro-2,8,8-trinitro-4,6-dioxanonane



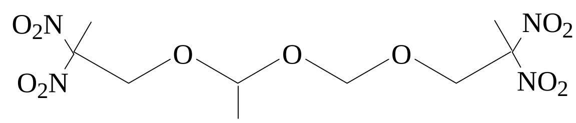
Compound 2

2-chloro-5-methyl-2,8,8-trinitro-4,6-dioxanonane



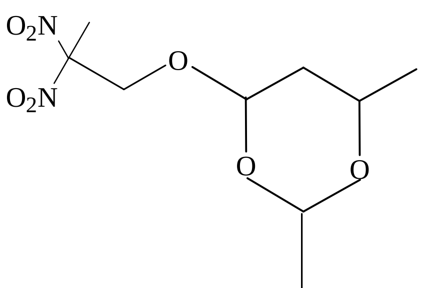
Compound 3

2,2,10,10-tetranitro-4,6,8-trioxaundecane



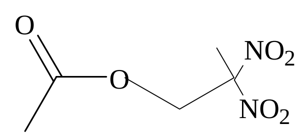
Compound 4

5-methyl-2,2,10,10-tetranitro-4,6,8-trioxaundecane



Compound 5

2,6,-dimethyl-4-[2,2-dinitropropoxy]-1,3-dioxane

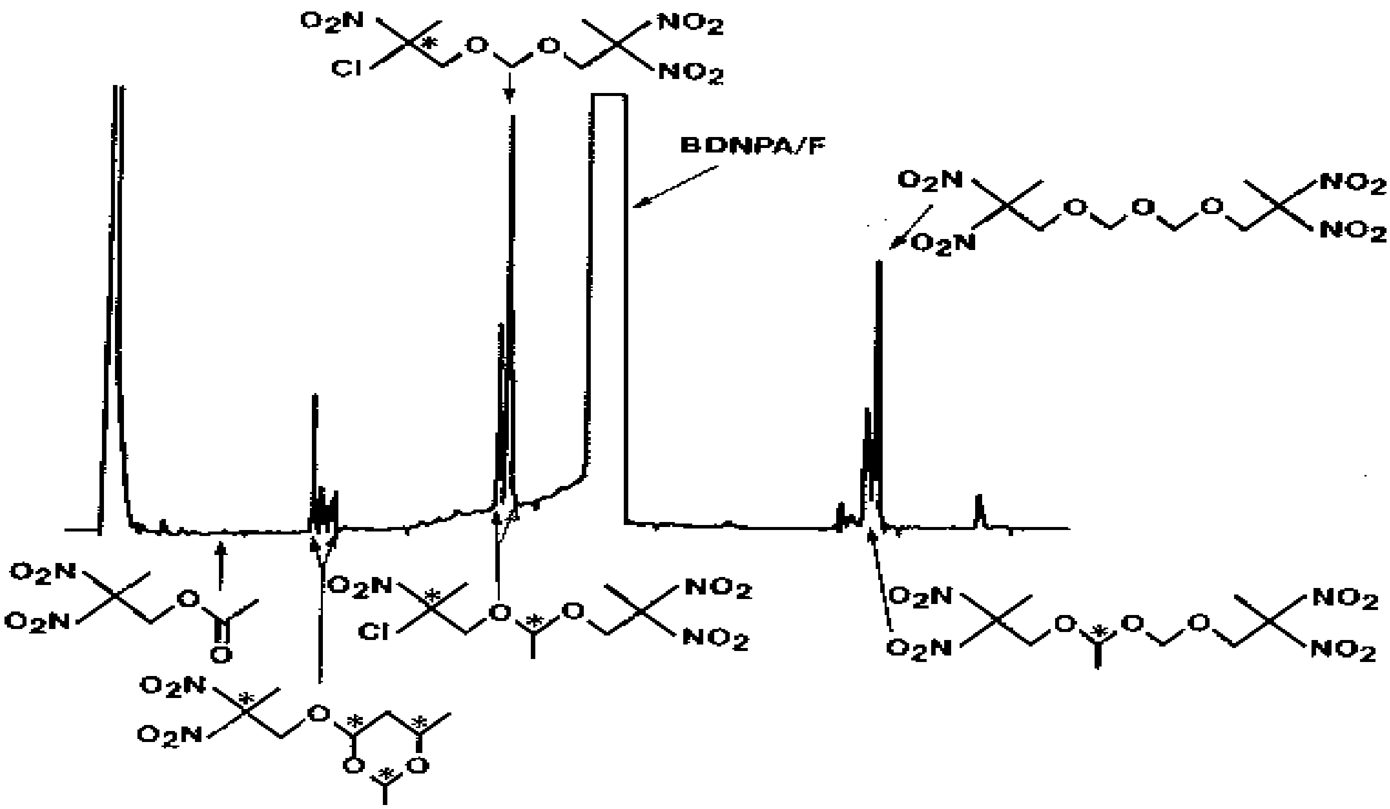


Compound 6

2,2-dinitropropyl acetate

FIGURE IV

A/F GLC ANALYSIS



Compound V

FIGURE V

CORRELATION OF INITIAL ACID NUMBER AND COMPOUND V CONTENT WITH PROBABILITY OF PASSING THE 1 WEEK ACCELERATED AGING TEST AT 105° C

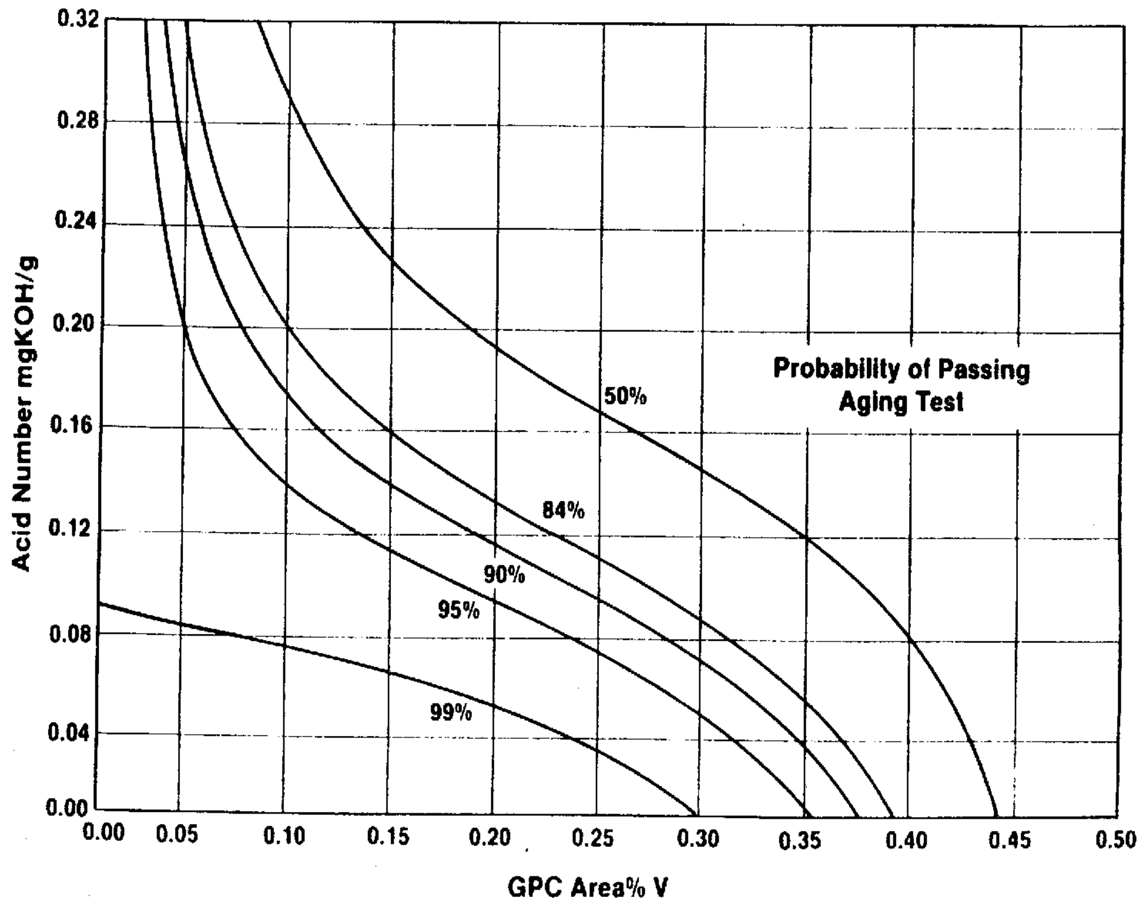
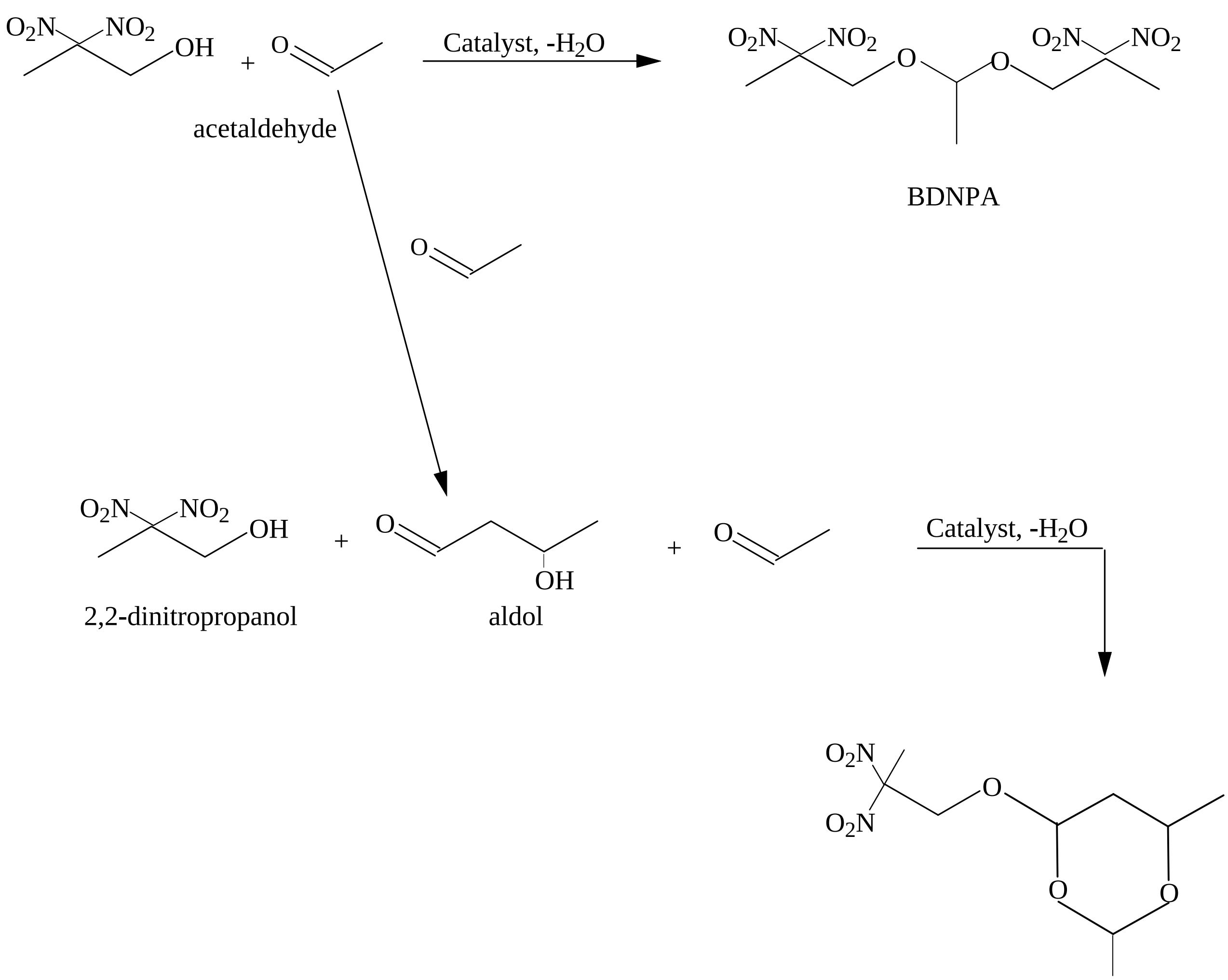


FIGURE VI

FORMATION OF 2,6-DIMETHYL-4-(2,2-DINITROPROPOXYL)-1,3-DIOXANE (COMPOUND V) IN THE BDNPA REACTION

BDNPA Reaction



Compound V

The effect of Compound V on the aging stability was a major discovery. Knowing how this impurity was formed in the A/F process made it possible to understand how to remove it from the process. First, the specifications on acetaldehyde were controlled more strictly. Aldol content, which was not considered in the original acceptance specification for the early A/F production, was now controlled to low levels in the raw material. Second, by closer control of the BDNPA production, Compound V was nearly eliminated from the BDNPA process stream. The result of these two controls was the production of A/F with significantly improved aging stability.

Experimental information given to the Navy and to FluoroChem Corporation by Aerojet in 1990 showed when the early production runs of A/F (most likely containing considerable amounts of Compound V) were aged even at 82° C, high acid numbers resulted in just a few days. Comparatively, similar aging tests with the A/F made in later production (1980s), when the Compound V impurity was controlled to very low levels, showed that the A/F was very stable with little, if any, acid build-up. These data are shown in *Figure VII*.

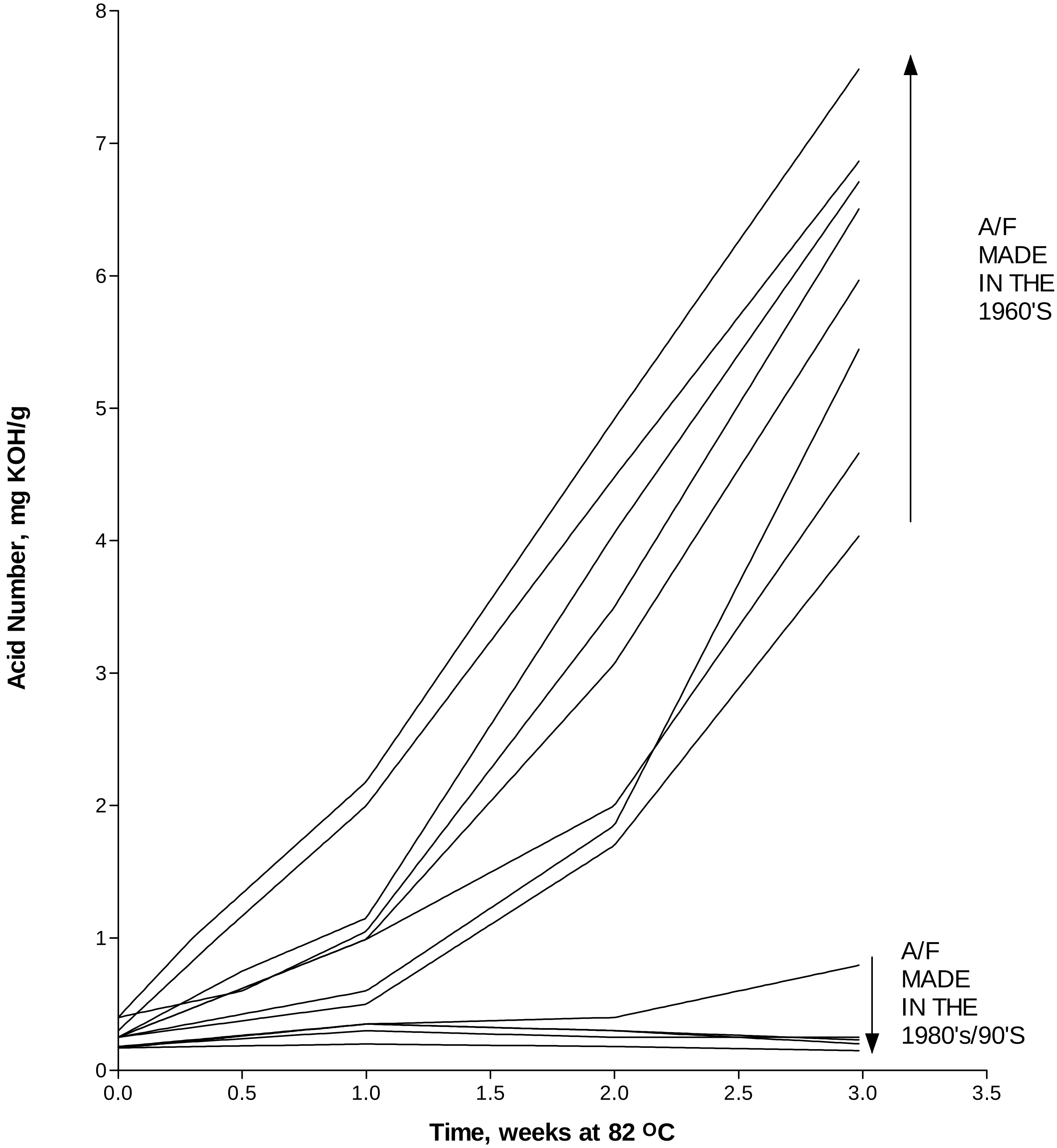
Additional A/F stability studies by the Navy conducted with Aerojet and Navy produced material also showed similar poor aging characteristics. As a result, the Navy created a new A/F specification test that would detect poor quality A/F. This test, referred to as the Accelerated Aging Test, required that A/F be heated at 104° C for a period of one week and not exceed the 0.5 mg KOH per gram of sample acid number. With this new test, A/F with poor aging or shelf-life characteristics would be revealed.

By identifying and removing the major process impurity (Compound V) in the improved A/F process, nearly every lot (i.e., approximately 250 lots) of A/F manufactured at Aerojet successfully passed the accelerated aging test. Upon implementation of the improved process, it was predicted (Arrhenius equation using accelerated aging data) that the A/F produced would have an extremely long (30 year estimate) shelf life if stored near 70 to 75° F but there was no actual shelf-life data to support this. That is until now.

Approximately 45 lots (150,000 pounds) of A/F produced at Aerojet in 1992 and 1993 were sold to Chugoku Kayaku Company, Ltd. (Chuka) in Japan. Over 100,000 pounds of A/F is still in Chuka's possession. This A/F was stored in a temperature-controlled room (about 72° F) for as long as eight years. Because Chuka has given DTCS exclusive resale rights for all A/F in their inventory, it was important to know how the A/F aged over this period of time. Were the earlier shelf-life predictions accurate? Was the A/F still in specification after being stored for seven to eight years? Recently

FIGURE VII

A COMPARISION OF AGING STABILITY OF A/F MADE IN THE 1960's VS. 1980's



(September, 2000), Chugoku Kayaku Co., Ltd (Chuka) provided DTCS with the answer. Ten Chuka lots (out of about 30 lots total) of their A/F were sampled at random and analyzed by Chuka using methods described in WS 1141A and its improvements, including the accelerated aging test at 104 to 105° C for one week. Without exception, every A/F lot tested passed the original specification tests. Most importantly, every lot of A/F tested passed the accelerated aging test with very little change in acid number when compared to the original test data at the time the A/F was manufactured. These data are shown in *Tables I and II* and *Figure VIII*.

The change in acid number of A/F stored over the seven to eight years was found to be very small. These data show that by reducing the concentration of Compound V in the A/F, the quality of the A/F remains intact. It follows that when this A/F is used in various propellant and explosive formulations, a significant improvement in the shelf-life is also expected.

TABLE I

COMPARITIVE ANALYSIS OF AGED BIS(2,2-DINITROPROPYL)ACETAL AND FORMAL (A/F)

Lot No.	Date of Analysis	Acid Number mg, KOH/g	Aged Acid No. mg, KOH/g	Water content wt. %	Refractive Index, nD 25C	Density g/ml, 25C	BDNPA wt. %	BDNPF wt. %	Stabilizer DPA, wt. %
2R-176-058	09/06/2000	0.17	0.20	0.044	1.4637	1.394	50.4	49.5	0.082
	07/31/1991	0.12	0.18	0.010	1.463	1.394	50.4	49.5	0.12
2R-176-073	09/06/2000	0.15	0.19	0.035	1.4635	1.391	51.4	48.5	0.097
	10/14/1991	0.12	0.18	0.006	1.464	1.392	51.5	48.5	0.13
2R-176-085	09/06/2000	0.10	0.15	0.028	1.4636	1.394	46.6	49.0	0.100
	02/07/1992	0.07	0.13	0.007	1.464	1.390	46.7	49.0	0.13
2R-176-088	09/06/2000	0.10	0.14	0.023	1.4635	1.391	48.6	48.9	0.093
	02/07/1992	0.07	0.15	0.006	1.464	1.391	48.7	48.8	0.13
2S-176-136	09/06/2000	0.16	0.18	0.030	1.4636	1.394	48.4	48.5	0.083
	05/04/1992	0.12	0.13	0.010	1.464	1.392	48.4	48.4	0.15
2S-176-138	09/06/2000	0.13	0.15	0.024	1.4634	1.390	49.1	48.9	0.096
	05/04/1992	0.11	0.13	0.006	1.463	1.391	49.3	48.9	0.14
2S-176-142	09/06/2000	0.17	0.20	0.044	1.463	1.398	47.9	48.2	0.091
	11/04/1992	0.09	0.17	0.008	1.464	1.394	47.9	48.3	0.14
2S-176-143	09/06/2000	0.17	0.27	0.038	1.4633	1.394	47.3	48.4	0.105
	11/04/1992	0.16	0.29	0.007	1.464	1.393	47.3	48.5	0.15
2S-176-144	09/06/2000	0.18	0.29	0.041	1.4634	1.391	47.6	45.8	0.085
	11/04/1992	0.12	0.31	0.010	1.463	1.393	47.7	45.5	0.14
2S-176-146	09/06/2000	0.18	0.26	0.033	1.4631	1.394	47.4	47.2	0.094
	02/02/1993	0.16	0.24	0.007	1.464	1.391	47.5	47.2	0.14
Specification Minimum		-	-	-	1.462	1.383	45	45	0.08
Specification Maximum		0.5	0.5	0.05	1.464	1.397	55	55	0.18

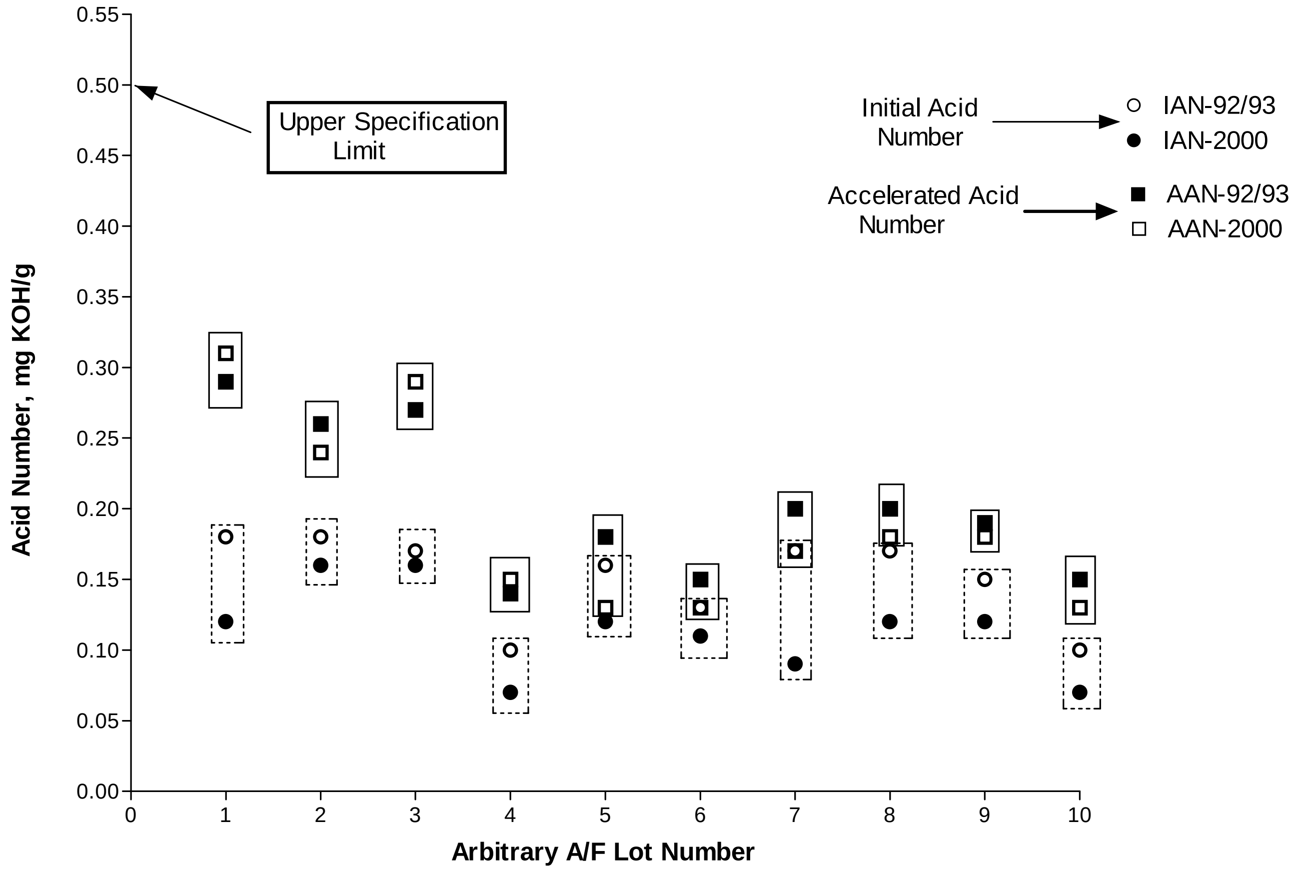
TABLE II

ACCELERATED AGING TEST ON SEVEN AND EIGHT YEAR OLD A/F

Lot No.	Date of Analysis	Initial Acid Number Mg, KOH/g	Acid Number After Accelerated Aging mg, KOH/g
2R-176-058	09/06/2000	0.17	0.20
	07/31/1991	0.12	0.18
2R-176-073	09/06/2000	0.15	0.19
	10/14/1991	0.12	0.18
2R-176-085	09/06/2000	0.10	0.15
	02/07/1992	0.07	0.13
2R-176-088	09/06/2000	0.10	0.14
	02/07/1992	0.07	0.15
2S-176-136	09/06/2000	0.16	0.18
	05/04/1992	0.12	0.13
2S-176-138	09/06/2000	0.13	0.15
	05/04/1992	0.11	0.13
2S-176-142	09/06/2000	0.17	0.20
	11/04/1992	0.09	0.17
2S-176-143	09/06/2000	0.17	0.27
	11/04/1992	0.16	0.29
2S-176-144	09/06/2000	0.18	0.29
	11/04/1992	0.12	0.31
2S-176-146	09/06/2000	0.18	0.26
	02/02/1993	0.16	0.24
Specification Minimum		-	-
Specification Maximum		0.5	0.5

FIGURE VIII

COMPARISON OF ACID NUMBER FOR 10 DIFFERENT LOTS OF UNAGED AND AGED A/F



A/F that has long-term storage stability is an important contribution in improving the producability and serviceability of the propellant and explosive formulations using it, such as PAX-2A[14,15,16,17,18].

PAX-2A has matured through extensive loading and testing to be a viable IM alternative to more sensitive HMX and RDX based explosives. Hazard Threat Assessment testing has been conducted with PAX-2A in various weapon system configurations. The sustained vulnerability level was proven to be either far superior or improved in comparison to most current fielded explosives. TACOM-ARDEC has also demonstrated that PAX-2A in Hellfire, Javelin, M830A1, SADARM, XM80 grenade submunition, and M77 grenade submunition warheads have met or exceeded penetration performance requirements.

Over 10,000 pounds of PAX-2A has been manufactured. PAX-2A has been made by TACOM-ARDEC, Holston-RONA, Thiokol Propulsion, Dyno, Hercules and Ensign Bickford. It has been demonstrated that this explosive can be loaded at full rate in high-speed loading production equipment.

PAX-2A has been selected for the 105mm M915 DPICM Projectile and PI SADARM submunition productions. The M915 projectile project management office has recently initiated a procurement request for 18,800 pounds of PAX-2A from Thiokol Propulsion. PM SADARM has acquired 2,000 pounds for future Product Improved (PI) SADARM production. PAX-2A is currently under consideration for many other weapon systems, including WAM, 155mm XM982 Excalibur, GMLRS and RADM.

Several future high energy PAX formulations based on CL-20 and A/F energetic plasticizer are currently being developed for use in the Army's Future Combat System (FCS).

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CONCLUSION

The impurities responsible for the formation of high acid concentration build-up during the storage of A/F have been identified. Neither BDNPA nor BDNPF decompose during storage and are not responsible for the formation of increasing acid concentration, as A/F is stored. Rather, Compound V, formed by the reaction of aldol with DNPOH in the BDNPA reaction, in the presence of catalytic amounts of acid impurities is responsible for long-term aging instability. A predictive tool has been developed that shows the relationship of beginning acid content and impurity Compound V concentration on overall A/F aging stability. This correlation gave conclusive evidence that A/F made with the improved “ter Meer” manufacturing process has an exceptionally long shelf-life, greater than experienced in the early production runs of A/F. Because there is no A/F storage stability problem as previously believed, A/F can confidently be used in continuing and developing new propellant and explosive formulations.